EFFECT OF COBALT OXIDE ADDITION ON ALUMINA SINTERING

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BY

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NATIONAL INSTITUTE OF TECHNOLOGY, ROURKELA
2015

NATIONAL INSTITUTE OF TECHNOLOGY, ROURKELA

CERTIFICATE

This is to certify that the thesis entitled, "**EFFECT OF COBALT OXIDE ADDITION ON ALUMINA SINTERING"** submitted by **Mr. BISWAJIT M PATRA (111CR0572)** in partial fulfillment for the requirements for the award of Bachelor of Technology degree in Ceramic Engineering at National Institute of Technology, Rourkela is an authentic work carried out by him under my supervision and guidance.

To the best of my knowledge, the matter embodied in this thesis has not been submitted to any other University/Institute for the award of any Degree or Diploma.

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CONTENTS

ABSTRACT

The present work describes the effect of cobalt oxide addition on alumina sintering. Here cobalt oxide is added as a dopant to enhance densification of alumina. In this project the dopant cobalt oxide is added by two ways firstly by direct addition of cobalt oxide to alumina by wet mixing and secondly by infiltration of cobalt nitrate solution into precalcined alumina pellets. In direct mixing process 1 and 2 mol% of cobalt oxide is added to alumina and in infiltration process infiltration is carried out for different time period such as 10, 20 ,30 and 40 minutes. Then the prepared pellets are dried and sintered at 1550^0C and 1600^0C with a soaking time for 3hrs. The pellets are then characterized for densification study, phase analysis and microstructural analysis. Final results reveal that the doped samples acquire a better density (~92% of theoretical density) than undoped samples (~ 86% of theoretical density) when sintered at 1600^0C.

INTRODUCTION

Alumina (Al_2O_3) is one of the most widely used ceramic materials. It has wide range of applications due to its excellent thermo mechanical, dielectric and other significant properties. It also has high compressive strength, high chemical resistance and it is transparent to microwave radio frequencies. It exists in several crystalline phases which all revert to the most stable alpha phase at elevated temperature. Alpha phase is the most stable and strongest phase of alumina. It has wide range of significant properties such as high refractoriness, high hardness, excellent dielectric properties etc. It's very good thermo mechanical properties are a result of close packing of aluminum and oxygen atoms in its crystal structure. Alpha alumina is also the most available and common crystalline form of alumina. It is also known as corundum. It has a trigonal Bravice lattice where each unit cell contains six formula units of aluminum oxide. The oxygen ions nearly form a hexagonal close-packed (HCP) structure with aluminum ions filling two-third of the octahedral interstices.

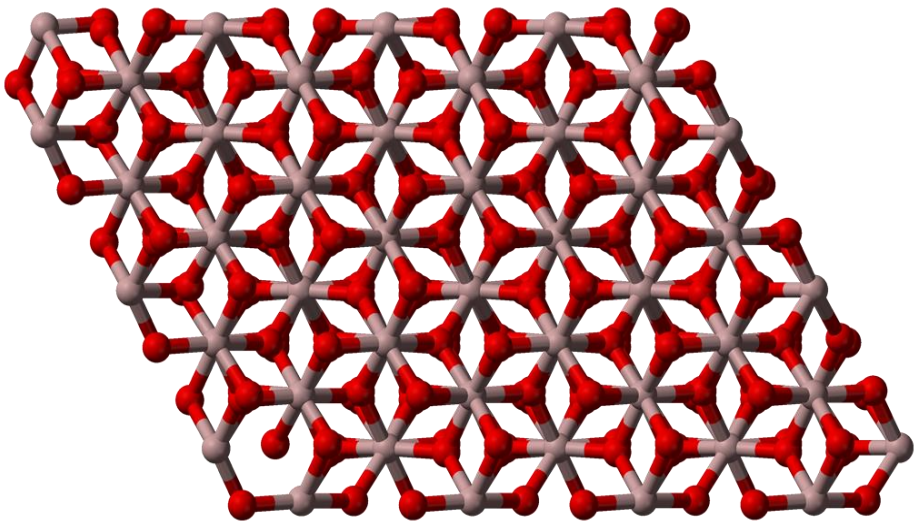


Fig-1: Crystal structure of alumina

Now a day there is a growing demand for ceramic materials with improved properties. The major use of alumina is in structural components where excellent mechanical properties are the major requirement. To obtain excellent mechanical properties the density should be as high as possible which can be done by optimizing the sintering parameters. This can be done mainly by two

ways. Firstly to optimize the fabrication parameters such as sintering temperature, time, particle morphology and granulometry. Secondly to design the microstructure by adding dopants to alumina so that higher density can be achieved at a lower temperature. There are some problems associated with processing of highly pure alumina. It requires much higher temperature and time for its sintering. To overcome this problem some dopants are added to alumina to decrease its sintering temperature and to increase its density without degrading its properties.

From various studies it is observed that MgO is the most common sintering additive added to alumina. It is expected that MgO decreases the grain boundary mobility of alumina thus preventing separation that effectively eliminates porosity and prevents abnormal grain growth and gives a higher densification. [1-2]

Many other dopants other than MgO have also been tried, but they are found to result abnormal grain growth thereby decreasing densification and other significant properties. Several recent studies also suggest that some rare earth metal and transition metal oxides can be used as sintering additive for alumina. [3-5]

The role of sintering additive is not only to enhance densification but also to provide uniform microstructure free from flaws so that the properties of the matter can also be improved. In the present work effect of cobalt (II) oxide as a sintering additive on alumina system is studied.

Fig-2: Structure of Cobalt (II) oxide

In the present work cobalt oxide is added to alumina by two methods, by direct mixing of solid oxides and by infiltration of dopant solution into the green alumina pellets. After that further processings and characterizations are done to study the densification behavior.

OBJECTIVE

The objectives of the present work are as follows:

- To study the densification behavior of cobalt oxide doped alumina samples at different temperatures
- To study the microstructure of cobalt oxide doped alumina samples at different temperatures
- To compare process of additive addition (direct addition and infiltration) for densification study.

LITERATURE REVIEW

SINTERING

Sintering is a densification process which generally occurs at higher temperature. It generally occurs by rearrangement, consolidation and diffusion of particles or atoms. Sintering can be classified into several types depending upon the mechanisms involved in densification or shrinkage. Sintering that proceeds mainly by solid state diffusion is known as solid state sintering whereas the sintering in which considerable amount of liquid is involved in densification process is said to be liquid phase sintering. The entire sintering process occurs in 3 stages such as:

(i) Initial stage: In this stage rearrangement of particles occurs mostly. Particles attain most stable position by rotating and sliding in response to the sintering force.

(ii) Intermediate stage: In this stage pores attain their equilibrium shape. Densification occurs by reduction in cross-section of pores.

(iii) Final stage: In this stage elimination of isolated pores occurs until maximum possible density is achieved.

Sintering is carried out by several mechanisms stated below

- Surface diffusion
- Lattice diffusion from grain boundary
- Lattice diffusion from the surface
- Grain boundary diffusion
- Vapour transport
- Viscous flow [6-10]

SINTERING OF ALUMINA BASED SYSTEMS

Emre Yalamaç, Antonio Trapani, Sedat Akkurt[11], prepared different batches of alumina by varying the proportion of alpha and gamma phase they sintered those batches at different heating rates of 1, 3.3 and 6.6 ^{0}C/min up to a constant temperature. They investigated the effect on sintering and microstructure of alumina by varying composition and heating rates. Finally they

concluded that the batch containing 10% gamma phase powder showed highest density for every heating rate.

Marco Cologna, John S.C. Francis, Rishi Raj [12], studied the Field assisted and flash sintering of MgO doped alumina and its relationship with conductivity. They reported that pure alumina with a nominal purity don't show field assisted sintering whereas MgO doped alumina show field assisted sintering due to larger conductivity provided by the ionic species. This ensures that MgO doped alumina shows higher conductivity of charged species so field assisted sintering occurs in that case.

EFFECT OF COBALT OXIDE ADDITION IN SINTERING

Kwang, **Min, Joo [13],** studied the effect of cobalt oxide addition on the sintering behavior of gadolinium aluminate ceramics. They found that by adding 5mol% cobalt oxide sintering temperature of gadolinium aluminate can be lowered by 150^0C. According to them the grain boundary layer melts at around 900^0C as melting temperature of cobalt oxide is around that temperature. The liquid phase formed in the grain boundary region helps in improving densification. It is also expected that due to high volatility of cobalt oxide, it can easily diffuse onto the surface matrix to form a thin amorphous film between the particles at low temperature to reduce friction thereby allowing viscous-flow sintering.

S.A Hassanzadeh [14] and others studied the effect of cobalt oxide addition on the sintering and grain growth of Al_2O_3-YAG nanopowder. From this experiment they found that cobalt oxide doping increased the ratio of densification to that of grain growth rate and also decreased the activation energy for grain growth from 590 to 485KJ/Mol thereby improving the densification significantly. This has happened due to small amount of cobalt oxide can lie within the solubility limit of the material so formation of solid solution leads to densification.

Eva Jud [15], and others studied the microstructure of cobalt oxide doped sintered ceria solutions. By looking at the microstructure the authors suggested that the cobalt oxide present within the ceria solutions in the triple points. At around 900^0C cobalt oxide diffuses into the grain boundaries where it forms a grain boundary film of thickness 0.5 nm thickness which promotes sintering at much lower temperature. Finally they concluded that by adding certain amount of cobalt oxide to ceria solution its sintering temperature can be decreased by 200^0C.

L.N. Satapathy [16], reported that cobalt oxide can be used as sintering additive for yttrium aluminum garnet. He reported that infiltration of 5% cobalt nitrate solution for 15min to a presintered YAG body enhances densification to a larger extent. He also suggested that cobalt nitrate melts at a lower temperature forming an amorphous grain boundary film which enables densification by limiting the abnormal grain growth.

A. Samson [17] and others reported that the infiltration of $La_{0.6}\,Sr_{0.4}CoO_{3-x}$ into Gd-doped ceria improves their performance to a significant level.

Rossi [18-19], and others studied the effect of additives on the microstructure of sintered alumina. They reported that additives used in alumina sintering limit the abnormal grain growth. In the grain growth process grains grow along with pores thereby abnormal grain growth induces residual pores in the microstructure and limits densification. As the additives limit abnormal grain growth they reduce entrapping of pores in the microstructure so they help in densification.

I-Wei Chen [20], reported how to control mobility of ceramic grain boundary and interfaces. According to him mobility of grain boundary and interfaces influences grain growth of ceramic materials largely which eventually influences the final density of the sintered material. Grain boundary and interface mobility of ceramic materials vary widely from material to material. In alumina grain boundary mobility is extremely sensitive to the presence of liquid phase and impurities.

EXPERIMENTAL WORK

RAW MATERIALS

In this experimental work mainly two raw materials are used. They are:-

Calcined Alumina

It is a form of alumina mainly consists of alpha phase of alumina. Due to excellent thermo mechanical properties it is mainly used for highly dense structural applications. Its chemical composition is as follows:-

Al_2O_3 - 94.14%

SiO_2 - 2.15%

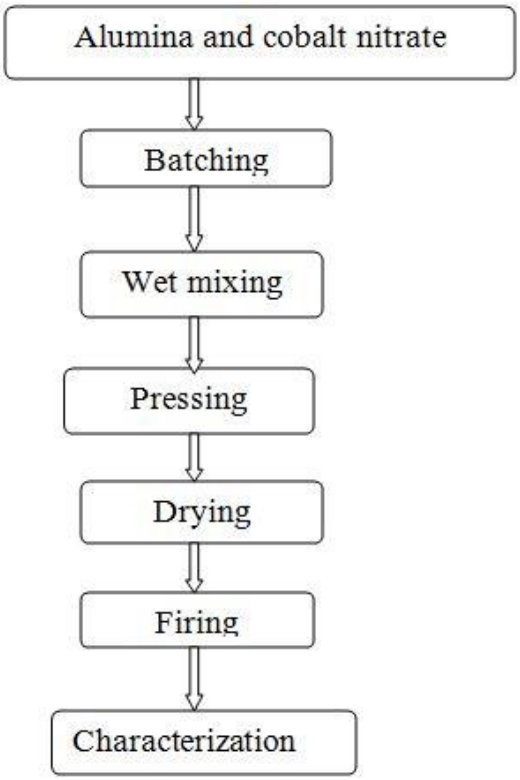
Fe_2O_3 – 1.78%

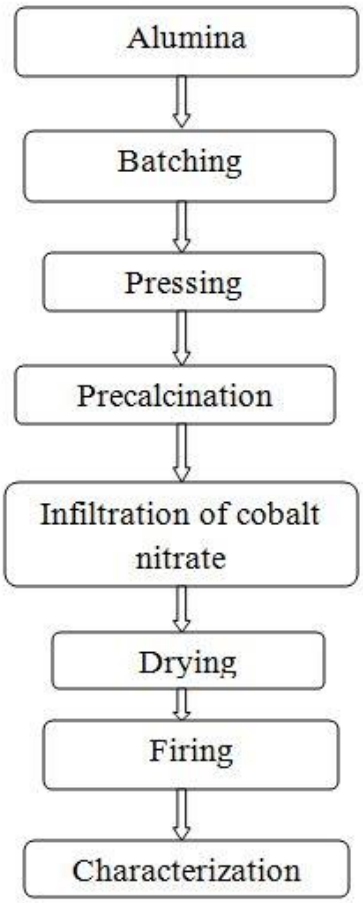
Cobalt Nitrate Hexahydrate

It is a salt of nitric acid having three dimensional polymeric network structures. It is soluble in water and other polar solvents. In this experiment cobalt nitrate hexahydrate ($Co(NO_3)_2.6H_2O$) is used as a source of Cobalt (II) oxide. Cobalt oxide is used as a sintering additive in this experimental work.

PROCESS FLOW CHART

In the present work cobalt oxide is added to alumina by two processes. The details of the processes are given below:





SAMPLE PREPARATION

(A)By direct mixing

Batching

Different batches are prepared by varying the amount of alumina and cobalt oxide. The actual batch composition is given in table1:-

Table-1: Batch composition of cobalt oxide directly added batches

Batch No	Alumina (Mol %)	Cobalt Oxide (Mol %)
Batch 1	100	0
Batch 2	99	1
Batch 3	98	2

Powder preparation

As the weight fraction of the dopants used is very low as shown previously so uniform mixing cannot be obtained via dry mixing. Mixing is done by wet mixing to obtain a uniform mixed composition. The batches are put in plastic jars, alumina balls are used as grinding media and distilled water is used as the liquid medium. Then the sample is allowed for pot milling for 24hrs in the liquid medium. After milling the mixtures are in the form of slurry are collected in Petri dishes. The slurry is dried at 120^0C for 24hrs to obtain powder from the slurry or to evaporate the moisture content. Then the obtained solid is grinded by the help of agitator and mortar to obtain free flowing powder.

Pressing

The obtained free flowing powder is to be pressed to make pellets. To increase the green strength of samples so that they can withstand further processing small amount of binder is added. Here

small amount of binder that is just sufficient to obtain green strength is added as dense ceramics is the ultimate desired product. Small amount of 2% PVA is added to the powder as binder and mixed properly with the help of agitator and mortar. The uniformly mixed powder is uniaxially pressed at 3ton load and 90 second dwell time. Here pellets of 15mm diameter are prepared from the powder with the help of 15mm die.

Drying & Firing

The green pellets are dried at 110^0C for 24hrs to attain sufficient green strength so that it can withstand further processing. The dried samples are fired at 1550^0C and 1600^0C with a soaking time of 3hrs at a heating rate of 3^0C/min.

(B)By infiltration technique

Powder preparation

First required amount of alumina powder is taken as per batch composition. Small amount of 2%PVA is added to alumina as binder to provide green strength. Then it is mixed uniformly to obtain a homogeneous composition.

Pressing

The obtained powder is uniaxially pressed in a 15mm die at 3ton load and 90 second dwell time. It is done to obtain green pellets of 15mm diameter.

Precalcination

It is done by firing the green pellets at 900^0C for 2hrs. It is done so that the green pellets would have sufficient strength to withstand the pressure during infiltration. It also provides another advantage that by this binder burns out so that it provides connected porosity for the dopants to uniformly distribute.

Infiltration

First infiltrations are tried by varying the infiltration time and concentration of cobalt nitrate in the solution. After studying obtained observations the final infiltration process is carried out in

5wt% cobalt nitrate solution. Infiltration process is carried out for samples of different precalcination temperature and for different time intervals. In this process the precalcined alumina pellets are put into cobalt nitrate solution kept in a beaker and the system is put in a vacuum desicator. Then vacuuming is done till desired time so that the solution should be infiltrated into the pellets. The solid loading of the dopant solution that is infiltrated is calculated by change in dry weight of the pellet before and after infiltration. Before infiltration weight of the sample is taken and after infiltration it is dried and weighed by subtracting the weights before and after infiltration solid loading is calculated. Different infiltrated batches are prepared by varying infiltration time and precalcination temperature while the keeping the dopant solution conc. constant.

Table-2: Batch composition of cobalt oxide infiltrated batches

Batch No	Precalcination temp(^{0}C)	Infiltration time (min)	Solid loading(in mol% of cobalt oxide)
Batch 4	900^{0}C	10	0.054
Batch 5	900^{0}C	20	0.058
Batch 6	900^{0}C	30	0.105
Batch 7	900^{0}C	40	0.106

Drying & Firing

After infiltration the pellets are dried at 110^0C for 24hrs to calculate the solid loading in infiltration. Then the dried samples are fired at 1550^0C and 1600^0C with a soaking time of 3hrs at a heating rate of 3^0C/min.

CHARACTERIZATION

XRD analysis

X-ray diffraction analysis is done for the fired samples to find the phases present in the final sintered product. It is carried out by using Rigaku Ultima-IV X-Ray diffractometer with nickel filtered Cu Kα radiation (λ=1.54A^o) and scanned in the range of 20^0 to 80^0. The XRD results of samples of different batches and sintered at different temperatures are analyzed to see the phases present in the fired product.

Volumetric Shrinkage calculation

The dimensions of the pellets are taken before and after firing. From the dimensions volume is calculated and volumetric shrinkage is calculated by the formula

Vol. Shrinkage= ((Vol. before sintering – Vol. after sintering)/ Volume before sintering)*100

Apparent porosity and Bulk density calculation

The apparent porosity and bulk density of sintered pellets are calculated by water boiling method. First dry weights of samples are measured and then they are put into a beaker and the beaker is filled with water. Then the beaker is heated for 45mins after boiling of water starts. After that the samples are allowed to cool and then soaked weight is taken. Then suspended weight is taken by suspending the sample in water. Hence the dry, soaked and suspended weights of each sample are obtained. Apparent porosity and bulk density are calculated using the following formula

Apparent porosity= (Soaked wt. - Dry wt.)/(Soaked wt. - Suspended wt.)*100

Bulk density= (Dry wt.)/ (Soaked wt. - Suspended wt.)

Field emission scanning electron microscopy

FESEM of the fracture surface of the fired pellets are done. It is done to reveal the detailed microstructure of the fired samples. FESEM (Field Emission Scanning Electron Microscope, Nova Nano SEM/FEI) instrument is used. The pellets are gold coated in a sputtering coater then they were loaded for analysis. EDAX is also done using the same instrument.

RESULTS & DISCUSSION

DILATOMETRY

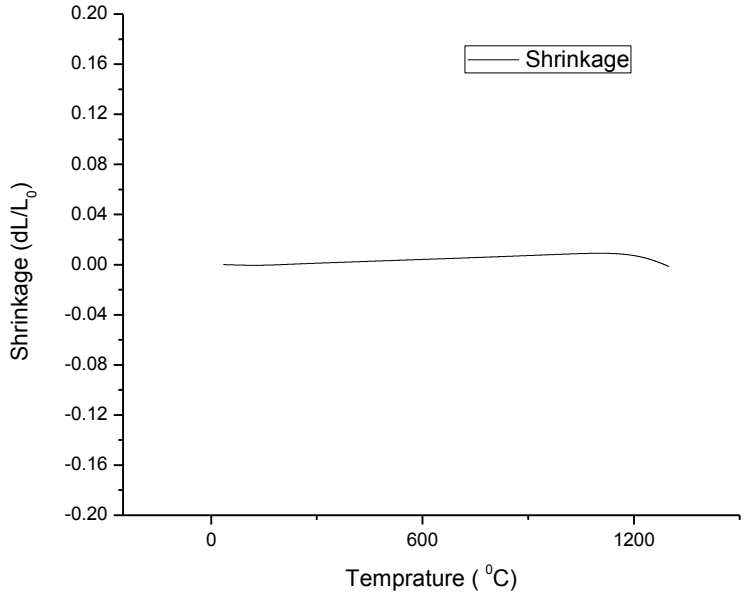


Fig-3: Plot for dilatomeric study of alumina

Fig-3 shows dilatometric study of alumina in the temperature range room temperature to 1300^0 C. The graph shows the variation in length of alumina sample with respect to temperature. From the graph it is observed that the sample expands at a slower rate in the low temperature range and this expansion continues up to around 1100^0C. This expansion occurs due to the decomposition of PVA and other volatile matters [3]. Above 1100^0 C shrinkage of the sample is initiated and that is due to starting of densification process of alumina sample.

APPARENT POROSITY

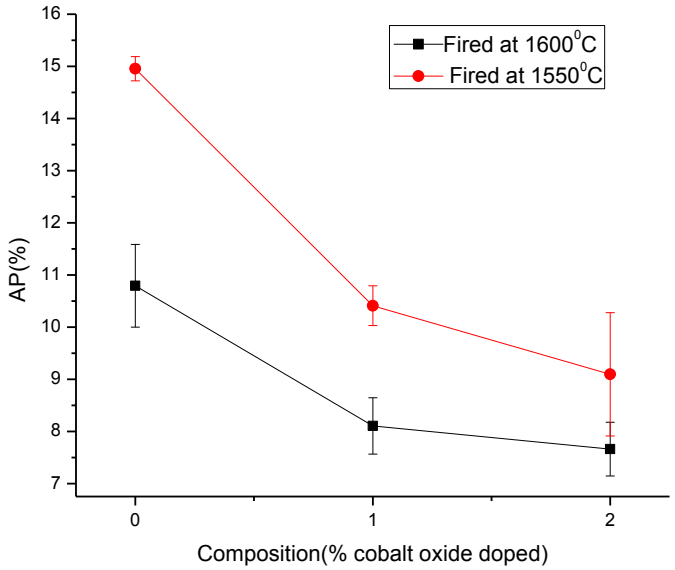


Fig-4: Plot for variation in apparent porosity of directly mixed batches

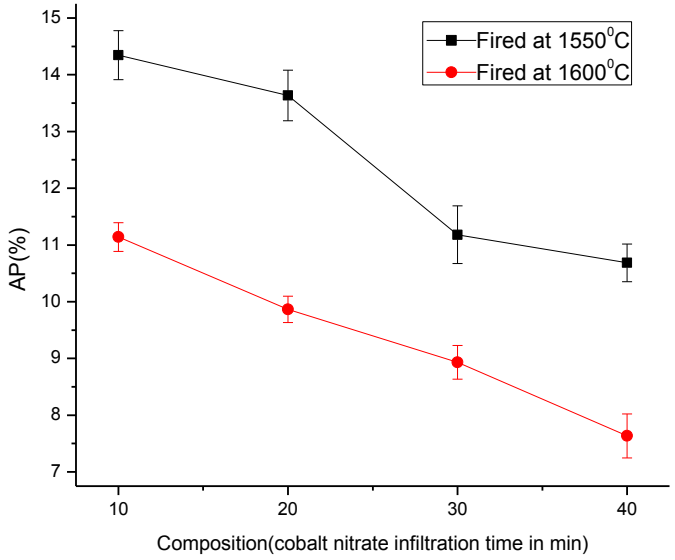


Fig-5: Plot for variation in apparent porosity of infiltrated batches

From fig-4 it is observed that apparent porosity of sintered alumina pellets decreases with increasing the amount of cobalt oxide. This is due to the sintering additive nature of cobalt oxide. It is also clear that with increasing sintering temperature porosity decreases due to increase in densification. The least porosity observed for directly mixed cobalt oxide is 8% for 2mol% cobalt oxide doped sample fired at 1600^0C. It is expected that the cobalt oxide will be present at the grain boundary thereby reducing the grain boundary mobility so less entrapped pores are present in the microstructure so porosity decreases with increase in cobalt oxide content as reported by L.N. Satapathy [16]. In case of cobalt nitrate infiltrated samples with increase in infiltration time amount of cobalt nitrate infiltrated is increasing so porosity is decreasing due to more densification. For infiltrated samples the samples precalcined at 900^0C then infiltrated for around 40 mins and fired at 1600^0C show lowest porosity around 7.5% as shown in fig-5.

BULK DENSITY

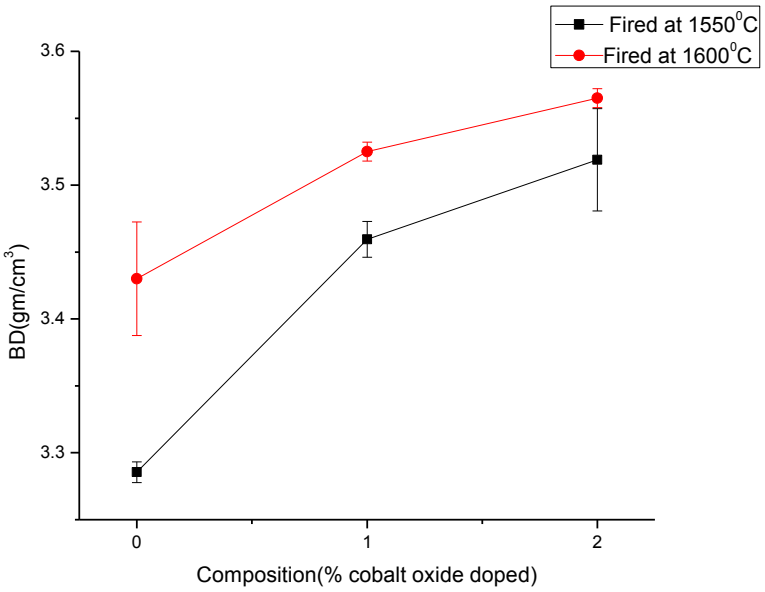


Fig-6: Plot for variation in bulk density of directly mixed batches

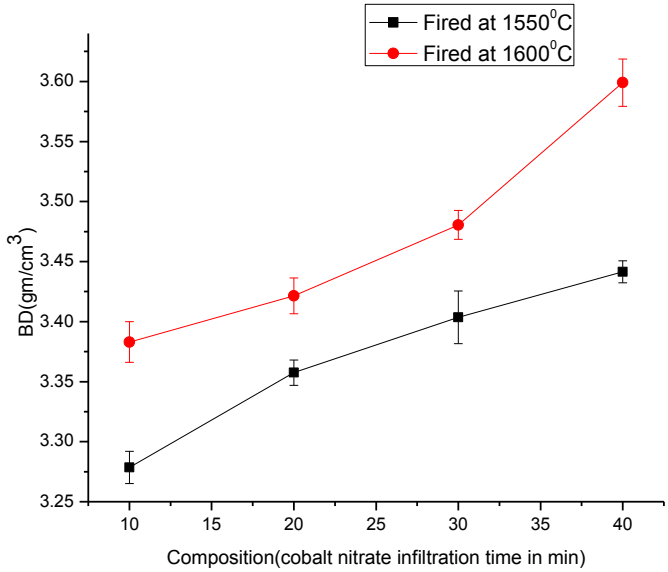


Fig-7: Plot for variation in bulk density of infiltrated batches

Fig-6 and Fig-7 show that bulk density of alumina pellets increases significantly with increase in amount of cobalt oxide and also with increase in temperature. Maximum bulk density achieved is around 92% theoretical density of alumina for the sample which is doped with 2mol% cobalt oxide and fired at 1600^0C and for samples precalcined at 900^0C then infiltrated for around 40 mins and fired at 1600^0C . Cobalt oxide is acting as a sintering additive which helps in densification of alumina at higher temperature by forming a low temperature liquid phase. With increase in amount of sintering additive apparent porosity is decreasing and bulk density is increasing simultaneously. Cobalt nitrate infiltrated samples are having almost same density as compared to direct cobalt nitrate mixed samples though the amount of cobalt nitrate added by infiltration process is very less (0.05-0.2 mol%) which is very less to direct mixed ones. With very little amount of addition of sintering additive by infiltration process helps the densification process to a much larger extent this is due to the additive added by infiltration process gets uniformly distributed in the microstructure which helps in sintering.

SHRINKAGE

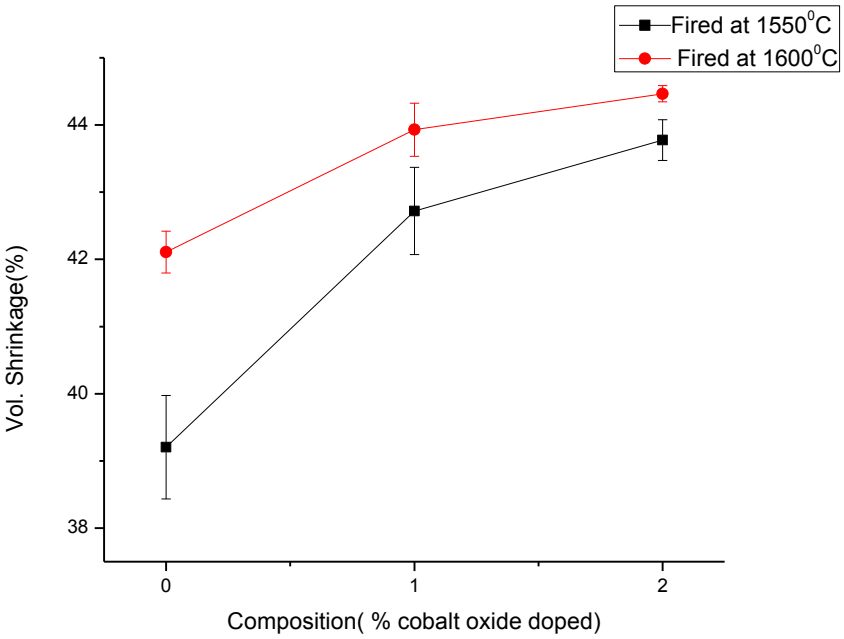


Fig-8: Plot for variation in shrinkage of directly mixed batches

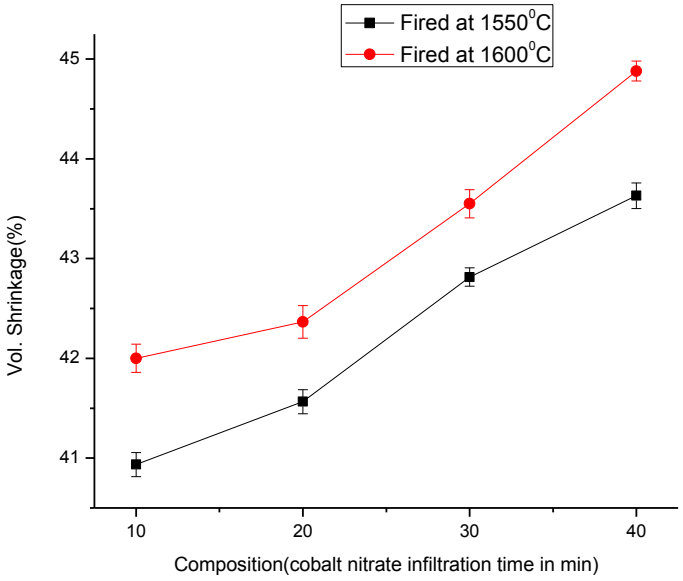


Fig-9: Plot for variation in shrinkage of infiltrated batches

Volumetric shrinkage of sintered pellets increases with increase in cobalt oxide content for the directly mixed batches and with increase in infiltration time for the infiltrated batches as shown in fig-8 and fig-9. Cobalt oxide acts as a sintering additive enhances densification so it causes more shrinkage to obtain a more dense body. As less entrapped pores will be present in the microstructure so more shrinkage occurs with increasing the amount of sintering additive. The maximum volumetric shrinkage obtained is around 45% for the samples precalcined at 900^0C then infiltrated for around 40 mins and fired at 1600^0C. The maximum shrinkage of these samples can be explained by the uniformity in distribution of cobalt oxide in the microstructure.

XRD ANALYSIS

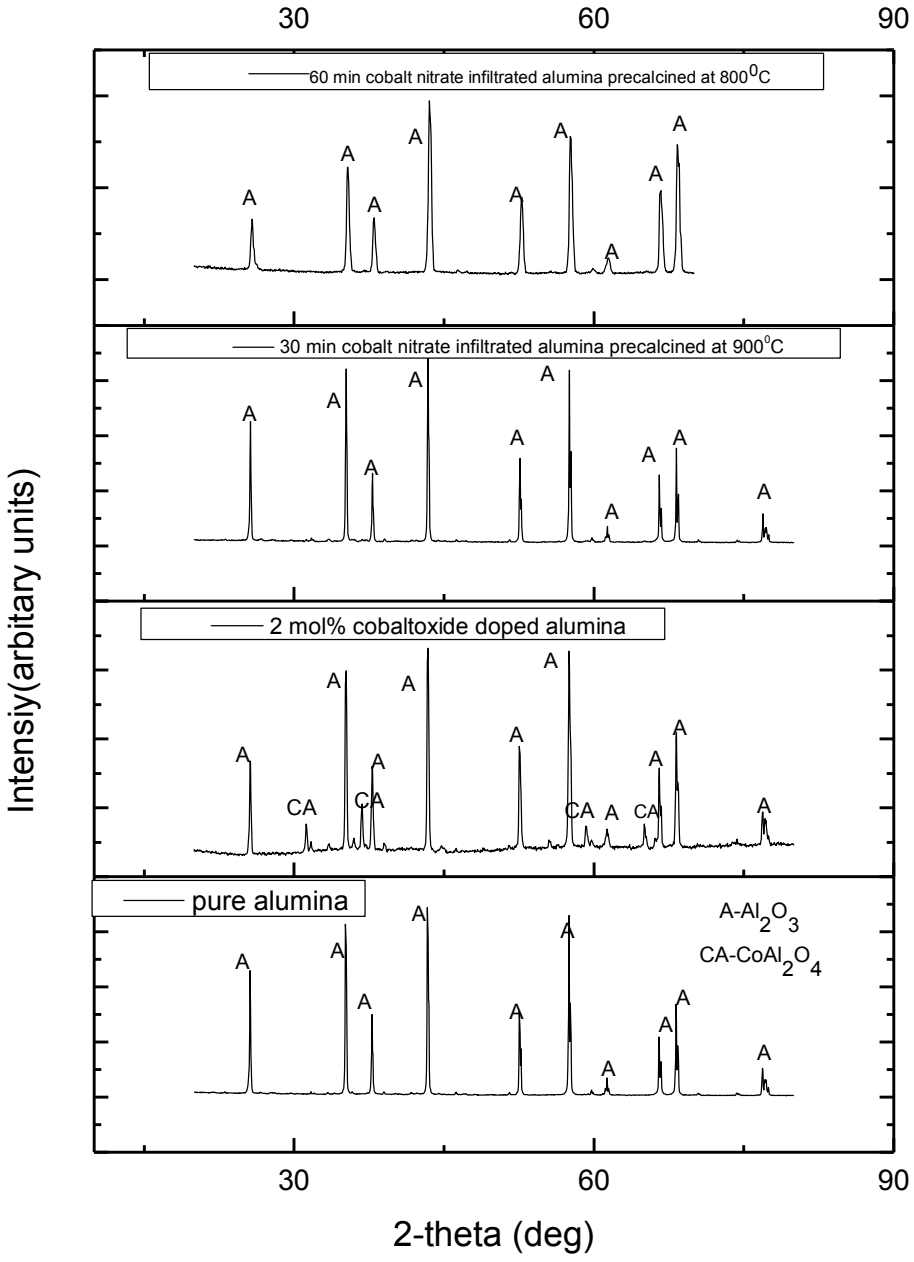


Fig-10: XRD analysis of sintered samples

From Fig-10 it is observed that only alumina peaks are detected in all batches except for 2mol% cobalt oxide doped batch.For this batch mostly alumina peaks and very small cobalt aluminate peaks are detected which conform that small amount of cobalt aluminate is formed during firing of the batch. For infiltrated samples cobalt oxide present acts as a sintering additive which enhances densification to a much larger extent, but their peaks are not dected in the XRD plot. This is due to the presence of sintering additive is very less around (0.05-0.2) mol%. No new phase formation is observed in case of cobalt nitrate infiltrated batches. Cobalt oxide melts around 900^0C and coats the grains of alumina so helps in liquid phase sintering.

FESEM & EDAX ANALYSIS

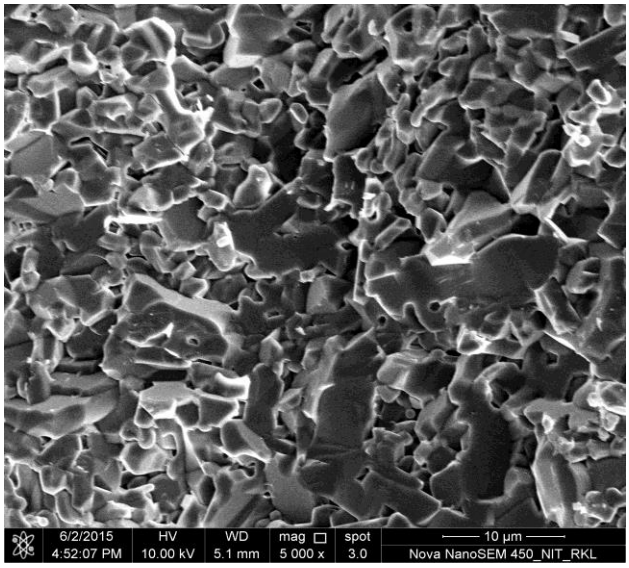


Fig-11: FESEM micrograph of pure alumina fired at 1600^0C

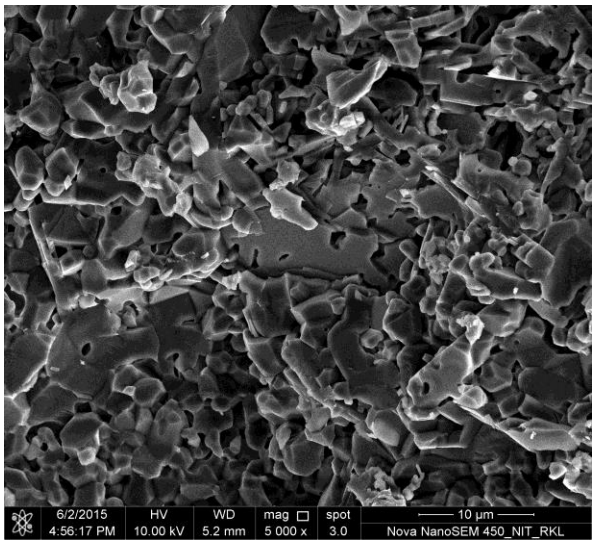


Fig-12: FESEM micrograph of 2mol% cobalt oxide doped alumina fired at 1600^0C

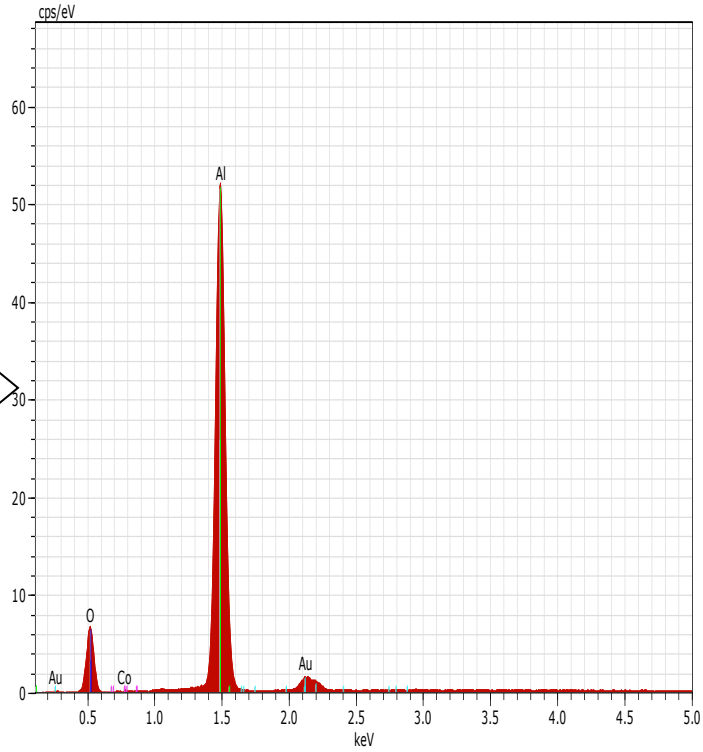
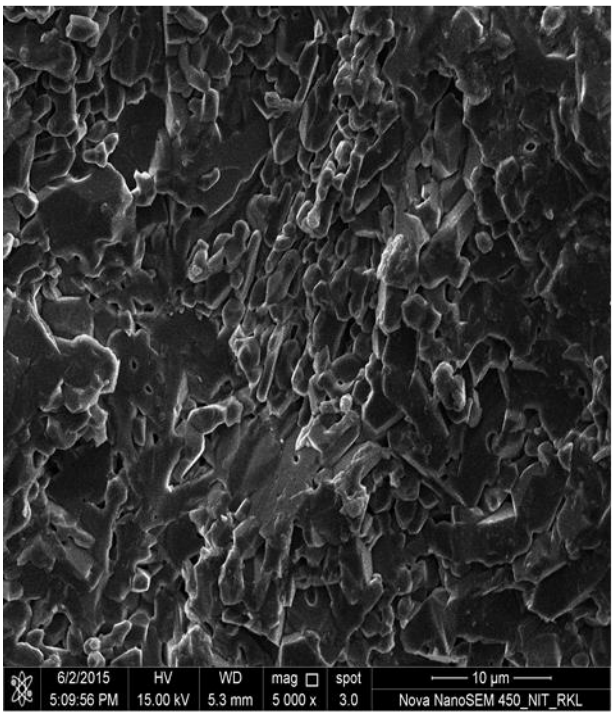


Fig13- EDAX image of 2mol% cobalt oxide doped alumina fired at 1600^0C



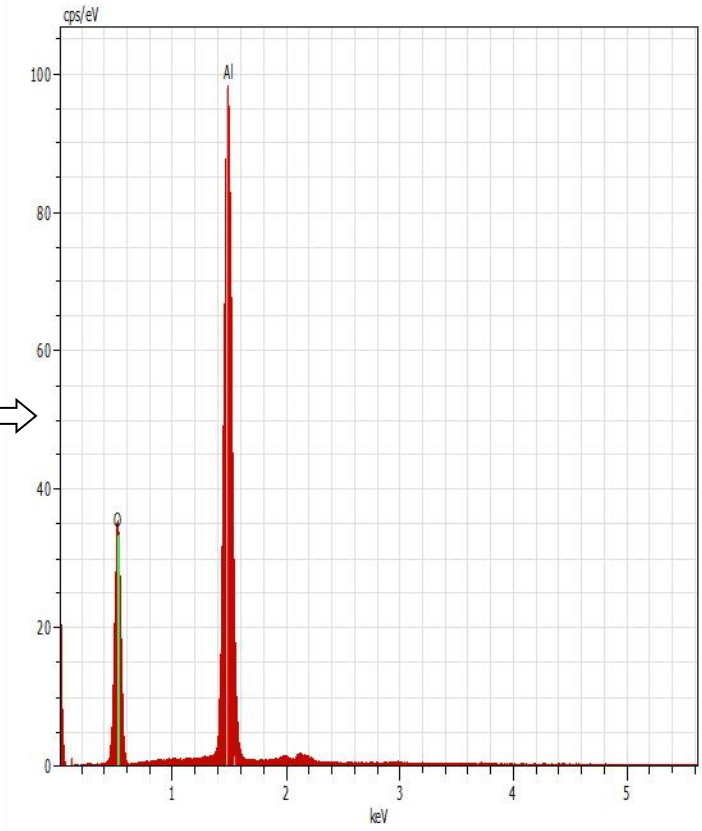


Fig-14: FESEM micrograph of 900^0C precalcined 40mins cobalt nitrate infiltrated alumina fired at 1600^0C

Fig-15: EDAX image of 900^0C precalcined 40 mins cobalt nitrate infiltrated alumina fired at 1600^0C

Fig-11,12 and 14 show the field emission scanning electron microscopy images of alumina and cobalt oxide doped alumina pellets sintered at 1600^0C for 3hrs. From the microstructure it is clear that more densification has occurred for cobalt oxide doped batches with less entrapped porosity as compared with undoped batch. For infiltrated samples grains are most densely packed. The infiltrated batch is having more dense microstructure as compared to directly added batch due to uniformity in presence of cobalt oxide in the microstructure. Fig-13 and 15 show that cobalt is present in cobalt oxide directly added batch but is not present in infiltrated batch. This implies that in the infiltrated batch cobalt oxide is present as solid solution giving rise to highest densification.

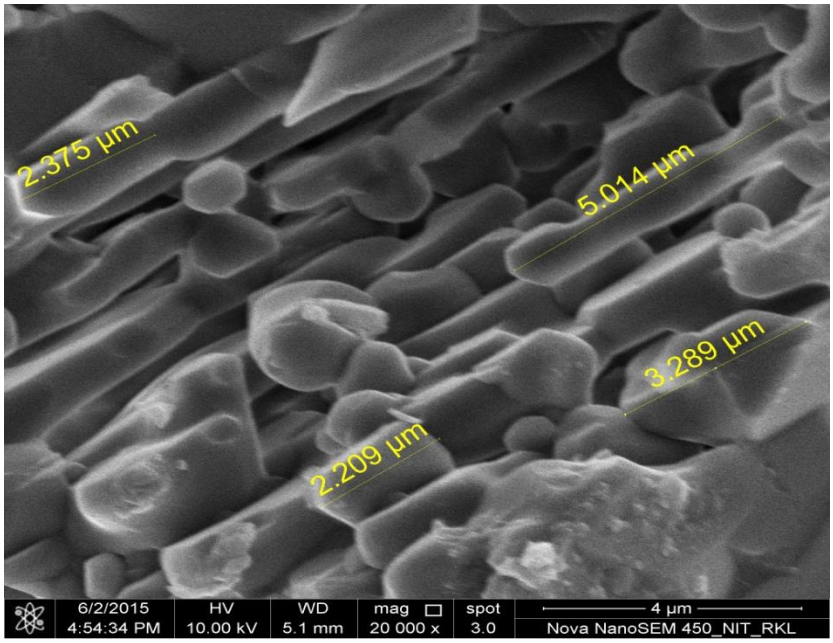


Fig-16: FESEM image showing grain size of pure alumina fired at 1600^0C

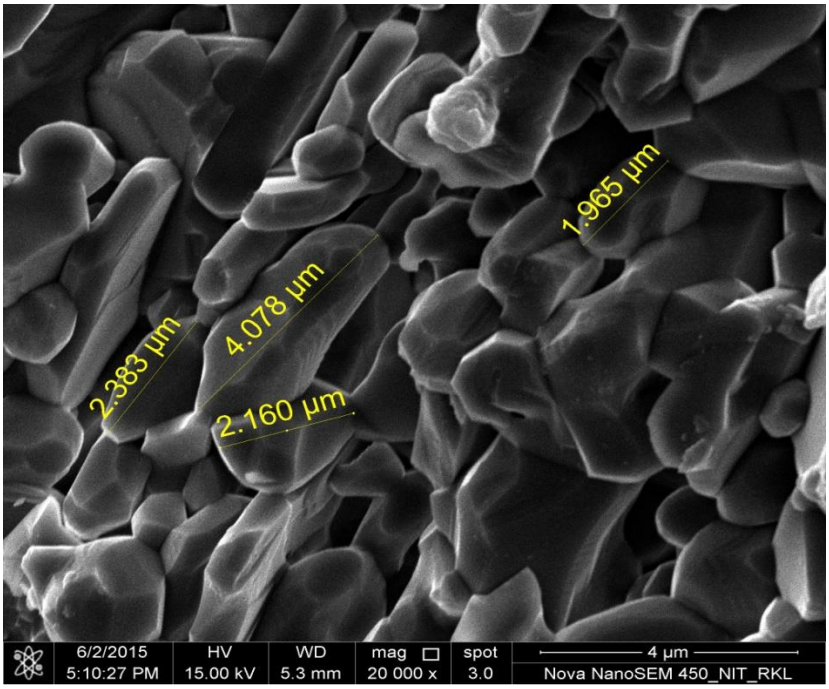


Fig-17: FESEM image showing grain size of 900^0C precalcined 40mins cobalt nitrate infiltrated alumina fired at 1600^0C

From fig-16 it is observed that abnormal grain growth took place in case of pure alumina pellets sintered at 1600^0 C. This abnormal grain growth is leading to grains of irregular shape and size. Due to irregular shape and size of grains more porosity is entrapped which leaded to limited densification. Fig -17 shows the microstructure of cobalt nitrate infiltrated and sintered samples where the grains are spherical in shape and of uniform size of length around 2μm. That means normal grain growth occurs in this case which leads to more densification as compared to pure alumina. This is due to cobalt oxide added by infiltration process is more uniformly distributed in the microstructure of alumina and its amount is very less. So no new phase formation occurs. The cobalt oxide present as solid solution melts at lower temperature which makes alumina grain boundary layers viscous so limits abnormal grain growth and enhance densification.

CONCLUSION

- Better densification results were obtained for cobalt oxide doped alumina samples than undoped samples. Thus bulk density, apparent porosity and shrinkage studies showed that cobalt oxide is effective as a dopant. A maximum densification of 92 % was obtained for doped samples fired at 1600 °C for 3 hrs.

- From the results it is also clear that cobalt oxide acts as a better sintering additive when infiltrated than direct addition because in case of infiltration 0.2mol% cobalt oxide providing same densification as provided by 2 mol% cobalt oxide when directly added.

- In cobalt oxide directly added batches cobalt aluminate phase forms in high temperature sintering but in case of cobalt oxide infiltrated batches no new phase formation occurs in sintering so cobalt oxide present in the microstructure enhance densification.

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